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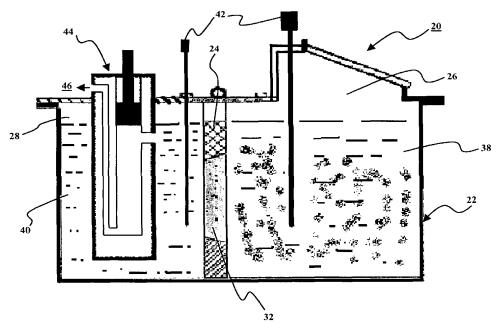
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[Continued on next page]

(54) Title: A FILTRATION SYSTEM FOR MAGNESIUM RECYCLING AND PURIFICATION



(57) **Abstract:** Magnesium alloys are heated to a molten state in preparation for hot-working thereof, for example, die-casting. The presence of inclusions within the magnesium alloys results in metallurgical defects therein and will adversely affect the quality of the castings produced from the magnesium alloys. An embodiment of the invention processes the magnesium melt containing impurities through a non-reactive filter under an environment filled with protective gas for substantially purifying the magnesium melt and to reduce the presence of inclusions in castings formed therefrom.



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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

1 A FILTRATION SYSTEM FOR MAGNESIUM RECYCLING AND PURIFICATION

Field Of Invention

The present invention relates generally to a filtration system for purifying magnesium. Specifically, the present invention relates to a filtration system for recycling and purification of scrap magnesium and magnesium ingots with impurities.

Background

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- Magnesium alloys are heated to a molten state in preparation for hot-working thereof. Molten magnesium alloys easily oxidise and react with impurities, especially when scrap magnesium alloys are reused. As a result, magnesium alloys are contaminated by non-metallic and metallic inclusions, for example oxides or intermetallic compounds, when melted.
- The magnesium alloys are typically melted for producing castings. The presence of inclusions within the magnesium alloys results in metallurgical defects therein and will adversely affect the quality of the castings produced from the magnesium alloys.
- A known method for removing the impurities is to send the magnesium alloys to a smelter for smelting. However, smelting is a costly process. Another known process uses impediment plates disposed within a furnace for removing top and bottom sludge from magnesium melts. However, the impediment plates do not remove inclusions suspended in the magnesium melts.
- Hence, this clearly affirms a need for a filtration system for purifying magnesium melts.

Summary

In accordance with a first aspect of the invention, there is disclosed a filtration system for magnesium recycling and purification, the filtration system comprising:

a first chamber for containing magnesium melt, the magnesium melt containing impurities; and

a second chamber for receiving purified magnesium melt,

wherein said first and second chambers have disposed therebetween a filter, the filter being a silicon-free medium.

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In accordance with a second aspect of the invention, there is disclosed a filtration method for magnesium recycling and purification, comprising the steps of:

receiving magnesium melt into a first chamber, the magnesium melt containing impurities within the first chamber containing impurities;

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providing a second chamber, the second chamber being in fluid communication with the first chamber; and

substantially removing the impurities from the magnesium melt flowing from the first chamber into the second chamber using a filter, the filter being disposed between the first chamber and the second chamber and the filter including a siliconfree medium.

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In accordance to a third aspect of the invention, there is disclosed a filtration method for magnesium recycling and purification, comprising the steps of:

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receiving magnesium into a first chamber, the magnesium within the first chamber containing impurities and the magnesium being one of a magnesium melt or solid magnesium ingot;

providing a second chamber, the second chamber being in fluid communication with the first chamber;

heating the magnesium contained in the first chamber by a heating apparatus for melting the magnesium and for maintaining the magnesium melt in a molten state, and the magnesium melt in the first chamber thereby flowing into the second chamber; and

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substantially removing the impurities from the magnesium melt flowing from the first chamber into the second chamber using a filter, the filter being disposed between the first chamber and the second chamber and the filter including a siliconfree medium.

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Brief Description Of The Drawings

Embodiments of the invention are described hereinafter with reference to the following drawings, in which:

- 10 FIG. 1 shows a partial front sectional view of a filtration system according to an embodiment of the invention;
 - FIG. 2 shows a partial side sectional view of the filtration system of FIG. 1;
- FIG. 3 is an illustration of a filter of the filtration system of FIG. 1;
 - FIG. 4 is an illustration of a first chamber and the filter of FIG. 3;
- FIG. 5a shows a low magnification light optical microscope (LOM) micrograph of a tensile specimen made from magnesium melt obtained from the filtration system of FIG. 1;
 - FIG. 5b shows the LOM micrograph of FIG. 5a under high magnification;
- FIG. 6a shows a low magnification light optical microscope (LOM) micrograph of a mobile phone case specimen made from magnesium melt obtained from the filtration system of FIG. 1;
 - FIG. 6b shows the LOM micrograph of FIG. 6b under high magnification;

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FIG. 7a shows a graph plotting the tensile strength (ultimate tensile strength and yield strength) of test samples as a function of cross-head speed with the test samples being cast from a magnesium melt purified using the filtration system of FIG. 1; and

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FIG. 7b shows a graph plotting the percentage elongation of the test samples of FIG. 7b, as a function of cross-head speed.

5 Detailed Description

An embodiment of the invention, a filtration system 20 is described with reference to FIG. 1, which shows a partial front sectional view of the filtration system 20 and FIG. 2 which shows a partial side sectional view of the filtration system 20. The filtration system 20 is for use in substantially purifying magnesium.

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As shown in FIG. 1, the filtration system 20 includes a crucible 22 being divided into two parts by a filter adapter 24 disposed therein, the two parts of the crucible being namely a first chamber 26 and a second chamber 28. The first chamber 26 is in fluid communication with the second chamber 28 through an opening 30, as shown in FIG.

2, in the filter adapter 24 that forms a passageway therebetween.

As shown in FIG. 2, the filter adapter 24 is for receiving a filter 32 therewithin and for removably engaging thereto. FIG. 3 is an illustration of the filter 32 and FIG. 4 is an illustration of the first chamber 26 and the filter 32. When the filter 32 is engaged to the filter adapter 24, the filter 32 blocks the opening 30 of the filter adapter 24, thereby intersecting the passageway between the first chamber 26 and the second chamber 28, as shown in FIGS. 1, 2 and 5. The filter adapter 24 is preferably a steel structure that shaped and dimensioned for holding the filter 32 at the periphery thereof.

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The filtration system 20 further includes a heating apparatus coupled to the crucible 22. The heating apparatus is preferably integrated with the crucible 22 for providing heat to the crucible 22 and its contents. The heating apparatus is electrically connected to a controller (all not shown).

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The filtration system 20 is for providing substantially purified magnesium melts to downstream systems or machineries, for example, a die-casting machine. For obtaining purified magnesium melts from the filtration system 20, magnesium ingots

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or scraps are provided to the first chamber 26 of the filtration system 20. The controller activates the heating apparatus to provide heat to the crucible 22 and the magnesium therein, thereby melting the magnesium.

Alternatively, magnesium melt can be provided to the first chamber 26 of the crucible 22. The heating apparatus provides heat to the crucible 22 to maintain the magnesium melt in its molten state and to melt the magnesium scraps and ingots added to the magnesium melt thereafter.

The filter 32 of the filtration system 20 is preferably made of a silicon-free material. Conventional filters, for example a filter for aluminium alloys, are made of silicon-based materials. The silicon-based materials readily react with magnesium to cause contamination therein and are therefore undesirable. The filter 32 is made of one of steels or ceramic material which comprises of one or more material selected from a group consisting of Al₂O₃, MgO, AlPO₄ and Mg₃(PO₄)₂.

The filter 32 comprises of an array of apertures (not shown). Each of the apertures is shaped and dimensioned for preventing the passage of a particle having a size greater than 5 microns therethrough. Preferably, each pair of adjacent apertures are spaced apart a distance of 5 to 250 microns. The magnesium melt 38 in the first chamber 26 passes through the filter 32 and into the second chamber 28 of the crucible 22. Therefore, the impurities suspended in the magnesium melt 38 contained in the first chamber 26 is substantially removed by the filter 32 before entering the second chamber 28 as purified magnesium melt 40. The magnesium melt 38 contained in the first chamber 26 contains bottom sludge that has settled at the bottom of the first chamber 26. In most situations, top sludge can also be found floating at the surface of the magnesium melt 38 contained in the first chamber 26. The filter adapter 24 functions to substantially impede the top sludge and bottom sludge in the first chamber 26 from entering the second chamber 28 (all not shown).

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With reference to FIG. 1, the magnesium melt 38 in the first chamber 26 is drawn into the second chamber 28 by hydrostatic forces acting on the magnesium melt 38. The magnesium melt 38 in the first chamber 26 continues to be drawn into the second

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chamber until the hydrostatic pressures of magnesium melt 38 in the first chamber 26 and the purified magnesium melt 40 in the second chamber 28 are in equilibrium.

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Preferably, each of the first chamber 26 and the second chamber 28 has a thermocouple 42 disposed therewithin. Both the thermocouples 42 are electrically connected to the controller for transducing temperature of the magnesium melt 38 in the first chamber 26 into first temperature signals (not shown) and the temperature of the purified magnesium melt 40 in the second chamber 40 into second temperature signals (not shown). The first and second temperature signals are transmitted to the controller. From the first and second temperature signals, the controller uses a control function (not shown) to determine and control the heat output of the heating apparatus, thereby maintaining the magnesium melt 38 and the purified magnesium melt 40 in a molten state and to prevent overheating thereof. In the molten state, the viscosities of both the magnesium melt 38 and the purified magnesium melt 40 are greatly reduced, thereby facilitating flow thereof through the filter 32.

The crucible 22 is preferably enclosed for receiving and retaining protective gas therein. A gas feed system (not shown) is connected to the crucible for supplying the protective gas thereinto. The protective gas prevents both the magnesium melt 38 and the purified magnesium melt 40 from reacting with the atmosphere by forming a screen therebetween.

An extractor 44, as shown in FIG. 1, extends from within the second chamber 28 to a die-casting assembly 46. The extractor 44 is for extracting the purified magnesium melt 40 from the second chamber 28 and providing the purified magnesium melt 40 to the die-casting assembly 46. The extractor 44 shown in FIG. 1 uses a piston and a goose-neck chamber assembly for extracting the purified magnesium melt 40.

Extracting the purified magnesium melt 40 from the second chamber 28 reduces the level of the purified magnesium melt 40 contained therein. The reduction of the level of the purified magnesium melt 40 in the second chamber 28 further draws the magnesium melt 38 from the first chamber 26 and into the second chamber 28. Magnesium melt and magnesium ingots or scraps can be further provided to the first

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chamber 26 for replenishing the second chamber 28 and thereby the filtration system 20 with purified magnesium melt 40.

The purified magnesium melt 40 supplied from the filtration system 20 to the diecasting assembly 46 provides the die-casting assembly with a substantially inclusionfree purified magnesium melt 40 supply for use in a die-casting process.

Two types of parts, hand-phone case and tensile test specimens, were cast during tests. The alloy used in the tests was AZ91 HP having a composition of Al 8-9.5%, Zn 0.3-1.0%, Mn \geq 0.17%, Si \leq 0.05%, Fe \leq 0.004%, Cu \leq 0.015%, Ni \leq 0.01%, others \leq 0.01%, others \leq 0.01%, Mg (remaining). For comparison the tests were conducted using 100% fresh ingot and ingot including 10% scraps material.

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After casting, the microstructure and chemical properties of the specimens were analyzed. The microstructure analysis was conducted with light optical microscopy (LOM) while mechanical properties were determined using an Instron tensile testing machine. The cross-head speed was varied from 0.1 to 20 mm/min during tensile tests.

Typical microstructures for the tensile test specimen, having a diameter of 10 mm-thick walled part and a mobile phone case, having a wall thickness of 0.6mm-thin wall part. In both thick and thin walled parts, the microstructures consist mainly of α-Mg, intermetallic-A1₂Mg₁₇, eutectic composition and some fine precipitate. However, the thin walled parts showed much finer structure, as shown in FIGS. 7a and 7b, when compared to the thick walled parts shown in FIGS. 6a and 6b. The reduced α-Mg grain size is due to rapid solidification rate occurred in the thin walled part.

Mechanical properties of cast samples were determined after casting. FIGS. 8a and 8b show the tensile strength, comprising the ultimate tensile strength (UTS) and yield strength (YS) and elongation as a function of the cross-head speed. As the cross-head speed is directly related to the strain rate of the testing, the results obtained indicated

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that the strain rate has a very slight influence on the flow stress and strain of magnesium castings when tested at room temperature. The UTS and YS are about 142 and 119 MPa respectively with an elongation of about 1%.

A comparison was made between the mechanical properties of castings made with filtered magnesium (with about 10% scraps) and castings made with fresh magnesium (100% new ingot). The strength of the castings is increased when filtered magnesium is used as compared to when 100% new magnesium ingot is used. However, the ductility of the magnesium alloys is reduced. The decreased ductility is typically due to the presence of more intermetallic compounds and less magnesium in the alloy having scrap parts therein although the alloy has already been filtered. Therefore, the composition of the alloy should be adjusted when using scrap parts.

In the foregoing manner, a filtration system is described according to an embodiment of the invention for addressing the foregoing disadvantages of conventional filtration devices. Although only one embodiment of the invention is disclosed, it will be apparent to one skilled in the art in view of this disclosure that numerous changes and/or modification can be made without departing from the scope and spirit of the invention.

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Claims

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- 1. A filtration system for magnesium recycling and purification, the filtration system comprising:
 - a first chamber for containing magnesium melt, the magnesium melt containing impurities; and
 - a second chamber for receiving purified magnesium melt,

wherein said first and second chambers have disposed therebetween a filter, the filter being a silicon-free medium.

- 10 2. The filtration system as in claim 1, the second chamber being in fluid communication with the first chamber and the filter for substantially removing the impurities from the magnesium melt flowing from the first chamber into the second chamber.
- 15 3. The filtration system as in claim 1, further comprising:
 - a heating apparatus integrated with the first and second chambers for maintaining the magnesium melt contained in the first and second chambers in a molten state.
- 20 4. The filtration system as in claim 1, further comprising:
 - a filter adapter disposed between the first chamber and the second chamber, the filter being removably coupled to the filter adapter and the filter adapter for positioning the filter to interface the first chamber and the second chamber.

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- 5. The filtration system as in claim 1, the filter comprising one of steel or ceramic material.
- 6. The filtration system as in claim 5, the filter further comprises at least one material selected from a group consisting of Al₂O₃, MgO, AlPO₄ and Mg₃(PO₄)₂.

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7. The filtration system as in claim 1, the filter comprising an array of at least one aperture.

- 8. The filtration system as in claim 7, each of the at least one aperture being shaped and dimension for preventing passage of a particle having a size greater than 5 microns therethrough.
 - 9. The filtration system as in claim 7, each pair adjacent apertures being spaced apart a distance of 5 to 250 microns.

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- 10. The filtration system as in claim 1, further comprising a temperature control system for regulating temperature of the magnesium melt within the first chamber and the second chamber.
- 15 11. The filtration method as in claim 10, the temperature control system comprising:
 - a controller, the heating apparatus being electrically connected to the controller;

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- a first thermocouple being electrically connected to the controller and being disposed within the first chamber for transducing temperature of the magnesium melt therein into first temperature signals; and
- a second thermocouple being electrically connected to the controller and being disposed within the second chamber for transducing temperature of the filtered magnesium melt therein into second temperature signals,

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wherein the first temperature signals and the second temperature signals are transmitted to the controller, the controller having a control function for controlling the heating apparatus and thereby maintaining the magnesium melt in a molten state and preventing overheating of the magnesium melt.

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12. The filtration system as in claim 1, further comprising a gas feed system for introducing a protective gas in to the first and second chambers.

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13. The filtration method as in claim 1, further comprising a step of:

an extractor for extracting the magnesium melt from the second chamber, the extractor being extending from within the second chamber to a die-casting assembly, the extractor for providing the extracted magnesium melt to the die-casting assembly.

14. A filtration method for magnesium recycling and purification, comprising the steps of:

receiving magnesium melt into a first chamber, the magnesium melt containing impurities within the first chamber containing impurities;

providing a second chamber, the second chamber being in fluid communication with the first chamber; and

substantially removing the impurities from the magnesium melt flowing from the first chamber into the second chamber using a filter, the filter being disposed between the first chamber and the second chamber and the filter including a silicon-free medium.

15. The filtration system as in claim 14, further comprising a step of:

providing a heating apparatus integrated with the first and second chambers for maintaining the magnesium melt contained in the first and second chambers in a molten state.

16. The filtration method as in claim 14, the filter comprising a silicon-free material.

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17. The filtration method as in claim 15, further comprising a step of:

providing a temperature control system for regulating temperature of the magnesium melt within the first chamber and the second chamber.

30 18. The filtration method as in claim 17, the temperature control system comprising:

a controller, the heating apparatus being electrically connected to the controller;

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a first thermocouple being electrically connected to the controller and being disposed within the first chamber for transducing temperature of the magnesium melt therein into first temperature signals; and

a second thermocouple being electrically connected to the controller and being disposed within the second chamber for transducing temperature of the filtered magnesium melt therein into second temperature signals,

wherein the first temperature signals and the second temperature signals are transmitted to the controller, the controller having a control function for controlling the heating apparatus and thereby maintaining the magnesium melt in a molten state and preventing overheating of the magnesium melt.

19. The filtration method as in claim 14, further comprising a step of:

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extracting the magnesium melt from the second chamber by an extractor, the extractor extending from within the second chamber to a diecasting assembly, the extractor for providing the extracted magnesium melt to the die-casting assembly.

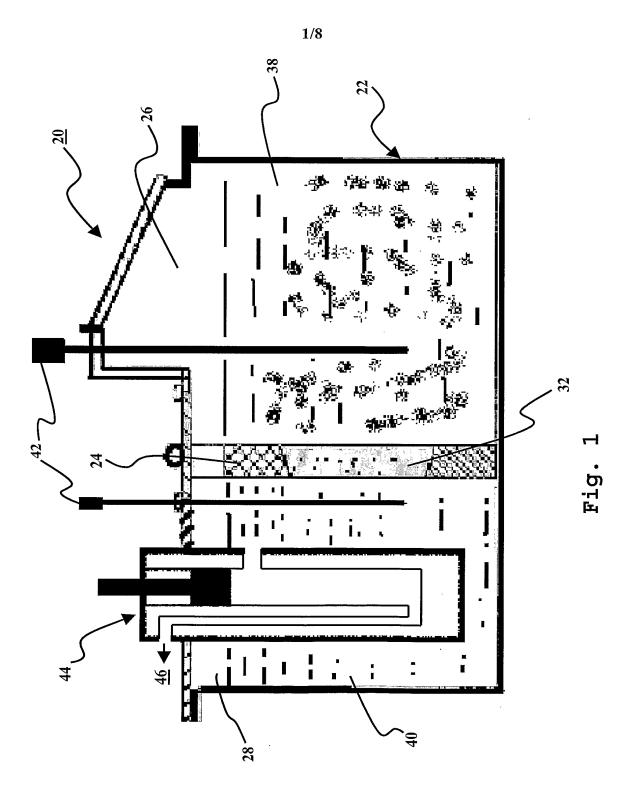
20. A filtration method for magnesium recycling and purification, comprising the steps of:

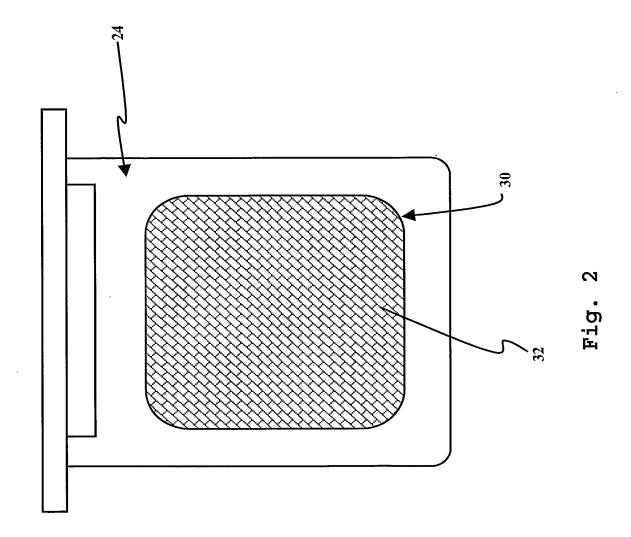
receiving magnesium into a first chamber, the magnesium within the first chamber containing impurities and the magnesium being one of a magnesium melt or solid magnesium ingot;

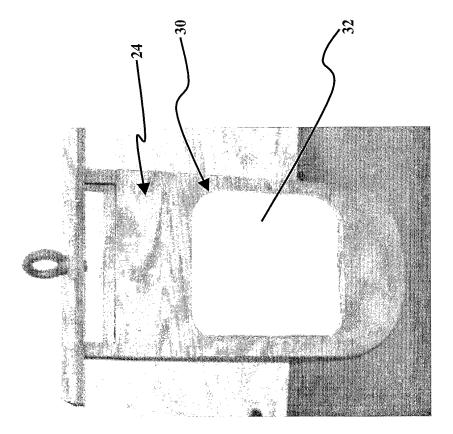
providing a second chamber, the second chamber being in fluid communication with the first chamber;

heating the magnesium contained in the first chamber by a heating apparatus for melting the magnesium and for maintaining the magnesium melt in a molten state, and the magnesium melt in the first chamber thereby flowing into the second chamber; and

substantially removing the impurities from the magnesium melt flowing from the first chamber into the second chamber using a filter, the filter being disposed between the first chamber and the second chamber and the filter including a silicon-free medium.







F19. 3

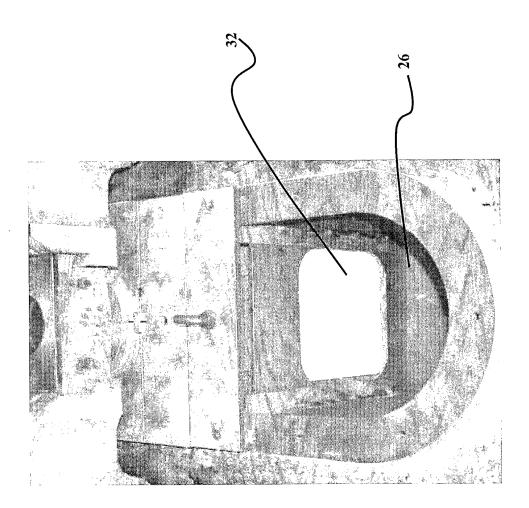
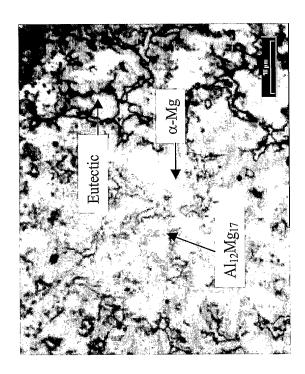


Fig. 4



lig. 5b

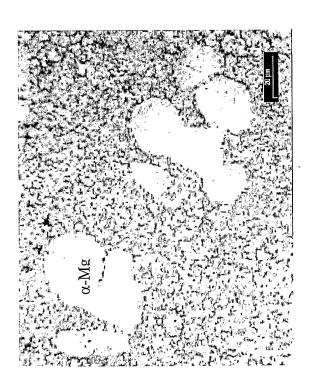
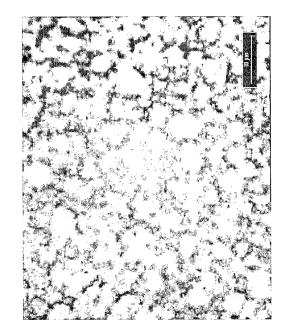


Fig. 5a

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ig. 6b

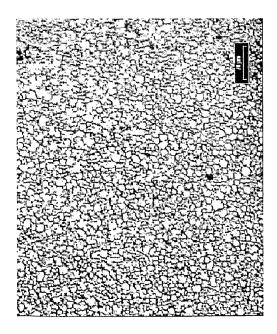


Fig. 6

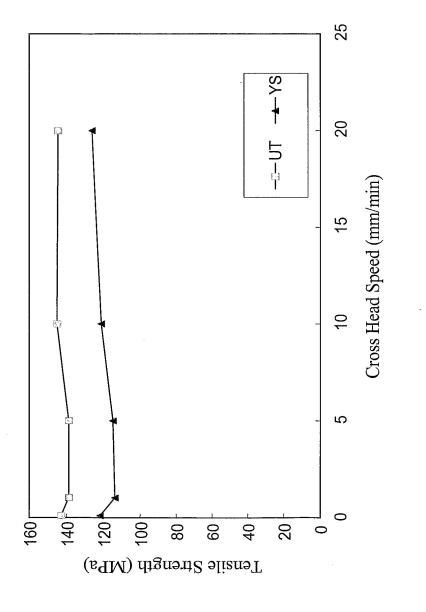
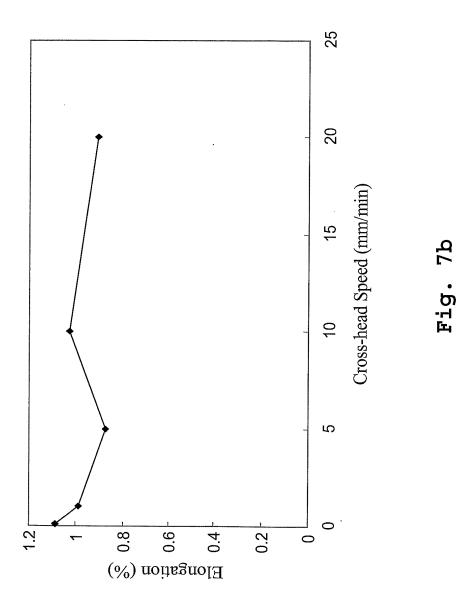


Fig. 7a



International application No. PCT/SG 02/00238

CLASSIFICATION OF SUBJECT MATTER

IPC7: C22B 9/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC⁷: C22B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI, PAJ

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